

**NBSIR 73-297**

# **Fracture and Deformation of Alumina**

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Final Report

Prepared for  
U.S. Army Research Office-Durham  
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U. S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary  
NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director



## FRACTURE AND DEFORMATION OF ALUMINA

### ABSTRACT

This report summarizes work conducted during the past eight years on the fracture and deformation of ceramic materials. Accomplishments discussed in this report include: an elucidation of fracture processes in aluminum oxide and sodium chloride; the development of techniques to study the deformation of aluminum oxide during abrasion; and the development of techniques for measuring fracture mechanics parameters on ceramic materials.



## FRACTURE AND DEFORMATION OF ALUMINA

This project was initiated to elucidate the mechanisms of fracture of brittle materials, with the hope of assuring the reliability of these materials in structural applications. Materials selected for study were aluminum oxide and sodium chloride. Aluminum oxide was selected because of its importance in ranging lasers, electronic substrates, ceramic armor and air bearings. Sodium chloride was selected because its mechanical properties were representative of a large group of ceramic materials having the sodium chloride structure. These include the alkali halides which today are important optical components of lasers.

Fracture mechanics techniques were used for these studies because of their advantage in elucidating fundamental processes occurring during fracture. Fracture mechanics techniques are used to measure the fracture surface energy or the stress intensity factor of a material. Since these are uniquely related to the crack tip stresses, they are the controlling mechanical parameters for crack extension. Thus by using fracture mechanics techniques one may relate various fracture phenomena, such as delayed failure, directly to the stresses at the crack tip.

The technique used for these studies was the double cantilever beam (DCB) technique developed by Gilman to measure the surface energies of single crystals. Because of the approximate nature of the equations used by Gilman, a complete elastic solution of the DCB configuration

was obtained in the course of the project.<sup>(2)</sup> This assured an accurate equation for calculating stress intensity factors from load and specimen dimensions. The complete elastic solution, obtained by the collocation procedure, agreed with the one used by Gilman for cracks that were long with respect to other specimen dimensions. However, for short cracks there were significant differences between the two solutions because of a contribution to the elastic energy from shear stresses in the crack arms and stresses in regions of the specimen past the crack tip. The elastic solution obtained in this program for the DCB configuration is now widely used by other investigators.

Initial experimental work revealed that the fracture of sodium chloride was severely influenced by plastic deformation at the crack tip, and that the fracture of sapphire was severely affected by water in the environment. Based on these observations, our experimental work developed along two lines: one, investigating the effect of environment on fracture; and the second, investigating the effect of plastic deformation.

Work on the plastic deformation of ionic crystals was conducted on sodium chloride. Two types of plastic deformation were observed at a crack tip, similar in many respects to plane stress and plane strain deformation observed in metals.<sup>(5)</sup> If plane stress deformation occurred, the crack intersected slip bands when it propagated and the energy for fracture was higher than the theoretical surface energy. However, if plane strain deformation occurred at the crack

tip, dislocation motion in the vicinity of the crack resulted in stress concentrations that aided crack motion. Consequently, the load for fracture, and the fracture surface energy were considerably less than predicted theoretically. These findings were consistent with a theoretical model proposed by Clark [Phil. Mag. 7, 393 (1962)] to explain observations of fracture in MgO, a material having the sodium chloride structure.

Environmental studies on single-crystal aluminum oxide (sapphire) indicated that the crack motion in this material is very similar to that occurring in glass. <sup>(1)</sup> Generally, fracture can be divided into three regions of behavior. At low stress intensity factors (region I) the crack velocity is influenced by the water in the environment and is strongly dependent on the applied load. At higher stress intensity factors (region II) crack motion depends only on the moisture in the environment. At still higher stress intensity factors (region III) the crack motion depends only on the stress and is independent of environment. This behavior parallels that observed on glass, and suggests that at all but the highest stress intensity factors, subcritical crack growth in sapphire is due to a stress corrosion reaction with water in the environment. These results agree with strength studies on polycrystalline alumina in which delayed failure has been attributed to moisture in the air. This delayed failure is undoubtedly due to the growth of cracks such as were observed in the current study.

This project was next directed towards an elucidation of the fracture process in sapphire. There is considerable controversy about the mechanism of fracture in this material. Because sapphire is very hard, exhibiting plastic flow only at temperatures greater than 900°C, it is expected to fail by brittle fracture at lower temperatures. However, its decrease in strength with temperature between 25 and 600°C is not consistent with the classical theories of brittle fracture. Therefore, some other explanation is necessary to account for the fracture behavior of sapphire. One group of investigators believes that the strength decrease results from a plastic flow process. They believe that fracture is intimately related to plastic flow and that as the temperature increases, the plastic flow and, consequently, fracture became easier. A second group disagrees with this conclusion suggesting instead that the reason for the strength decrease is that the fracture of sapphire is determined by thermally activated crack growth. It was our feeling that these opposing theories could be critically evaluated by studying the crack tip structure of sapphire using transmission electron microscopy, and by accurately characterizing the fracture behavior of this material via fracture mechanics techniques. Consequently, the fracture of sapphire was studied using fracture techniques developed earlier in this program. Also, new techniques were developed so that crack tips in sapphire could be examined by transmission electron microscopy.

Our studies of the fracture energy of sapphire can be divided into two parts: an evaluation of the fracture energy as a function of crystal

orientation; and an evaluation of the fracture energy and crack velocity as a function of temperature under vacuum conditions. Fracture energies as a function of crystal plane<sup>(3)</sup> were measured on three different planes of sapphire, the basal (0001) plane, the rhombohedral twin (01 $\bar{1}$ 2) plane, and the secondary prismatic (10 $\bar{1}$ 0) plane. Fracture energies for the rhombohedral and prismatic planes were 6.0 and 7.3 J/m<sup>2</sup>, respectively, but for the basal plane, fracture energies were estimated to be greater than 40 J/m<sup>2</sup>. Crack propagation on this plane by the double cantilever beam technique was not possible.

Fracture energy measurements were also made in vacuum by developing a new test apparatus capable of a vacuum of 10<sup>-5</sup> torr and of a temperature as high as 750°C.<sup>(12)</sup> Studies using this equipment showed that the fracture energy of sapphire (10 $\bar{1}$ 0 plane) depended on temperature, decreasing from 7.3 J/m<sup>2</sup> at room temperature to approximately 4.5 J/m<sup>2</sup> at 750°C. This change in fracture energy was consistent with strength measurements, suggesting that the strength decrease probably results from the effect of temperature on the crack propagation rate. It was also observed in this study that the temperature dependence of the fracture energy depended on the crystallographic orientation of the fracture plane, since cracks on the rhombohedral plane did not propagate on this plane at temperatures above approximately 200°C even though this was the favored plane at room temperature.

In another aspect of our work, new techniques of specimen preparation were developed for transmission electron microscopy studies of dislocation

interactions with crack tips.<sup>(6)</sup> The techniques developed for this study were based on earlier work at NBS by N. J. Tighe, who demonstrated the value of ion bombardment thinning as a method of specimen preparation. Specimens suitable for the electron microscope were prepared by first placing indentations in the surface of one-eighth inch sapphire or alumina disks. These indentations always initiated surface cracks which could be studied by transmission electron microscopy provided the specimens were thinned preferentially from the side of the disk opposite that containing the crack. Examination of these specimens revealed dense dislocation activity associated with the indentation.<sup>(8)</sup> However, little if any dislocation motion was associated with the crack tip.<sup>(12)</sup> This observation led to two additional studies in which the defect surface structure (cracks, slip bands and twins) formed during the grinding and polishing of sapphire was investigated.<sup>(7,10,11)</sup> Dislocation and twin rearrangement resulting from high temperature annealing were also studied.

Results from the transmission electron microscopy studies of crack tips indicated that little if any dislocation motion occurred when cracks were propagated at temperatures less than 400°C. Above 400°C, some dislocation motion was observed, but dislocation motion was the exception rather than the rule. Cracks that were propagated by mechanically stressing often healed spontaneously when the mechanical stresses were released. This type of healing occurred when two crack surfaces came back together in almost perfect registry. A different type of crack

healing occurred when specimens were annealed at high temperatures. In this case, healing occurred by diffusion and sintering of the crack tip. Pore growth and crack recession was always associated with crack healing at high temperatures. This high temperature annealing probably accounts for the increase observed in the strength of sapphire after annealing.

The most important result of the fracture and transmission electron microscopy studies is that dislocation motion does not account for the temperature dependence of the strength of sapphire. Strength degradation is more consistent with an activated crack growth process. However, the causes of this activated process are as yet ill-defined. Since the activation energy for the process is approximately 200 Kcal/mol, the fracture mechanism may involve oxygen or aluminum ion diffusion near the crack tip. Additional work will be necessary to further clarify the mechanism of fracture in this very important material.

In summary, work on this project has resulted in: the development of new techniques for determining fracture energy; a deeper understanding of the fracture process in alkali halide materials and sapphire; an exact elastic solution of the double cantilever beam configuration; the development of a new technique for studying surface damage in ceramic materials; and a characterization of surface damage resulting from the abrasive wear of alumina during polishing, grinding and abrasion operations. A list of all publications and technical reports published under Army Research auspices is given below.

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2. Critical Analysis of the Theory of the Double Cantilever Method of Measuring Fracture-Surface Energies, S. M. Wiederhorn, A. M. Shorb, and R. L. Moses, J. Appl. Phys. 39, 1569 (1968).
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4. Fracture of Ceramics, S. M. Wiederhorn, Proc. of Symposium on Mechanical and Thermal Properties of Ceramics, April 1-2, 1968, National Bureau of Standards, Washington, D. C., NBS Spec. Publ. 303, May 1969, pp. 217-41.
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7. Discussion of "Observation of Plastic Deformation in Sapphire Base Ceramics," A. H. Heuer, B. J. Hockey, and S. M. Wiederhorn, Metallurgical Transactions, 2, 1475 (1971).
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10. Observations on Mechanically Abraded Aluminum Oxide Crystals by Transmission Electron Microscopy, B. J. Hockey, Proc. of Symposium on "The Science of Ceramic Machining and Surface Finishing," NBS, Gaithersburg, Md., Nov. 2-4, 1970, NBS Spec. Publ. 348, May 1972, pp. 333-39.

11. Use of the Hardness Test in the Study of the Plastic Deformation of Single Crystals, B. J. Hockey, to be published in Proc. of Symposium on "The Science of Hardness Testing and Its Research Applications," Detroit, Mich., Oct. 18-21, 1971.
12. Effect of Temperature on the Fracture of Sapphire, S. M. Wiederhorn, B. J. Hockey, and D. E. Roberts, to be published in Phil. Mag.



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